ABSTRACT

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Title of Project:
Niobium Oxide-Metal Based Seals for High Temperature Applications
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The aim of this investigation is to establish the feasibility of using mixtures of Nb_2O_5 and Al_2O_3 powders to form a thermodynamically stable, high temperature seal between alumina and other materials for gas separation applications. Several composition ranges have been prepared and examined with x-ray diffraction and electron microscopy. Results to date are encouraging in that it appears that for certain compositions, a glassy phase that wets Al_2O_3 forms at around 1400ºC or below.

Powders of Nb_2O_5 and Al_2O_3 were mixed in several different composition ranges, and either hot pressed or sintered. Oxidation pretreatments of Nb powder to form Nb_2O_5 were planned, but preliminary experiments revealed that this did not provide sufficient control over the quality and composition of the oxide powder, and thus Nb_2O_5 was purchased directly from a chemical supplier. Specimens of the following mol. % Nb_2O_5 were fabricated by cold pressing and sintering to approximately 1400ºC: 52, 60, 67, and 88. These were chosen to explore composition ranges that experience different amounts of the eutectic composition. Porosities measured by the Archimedes method ranged from 4 to 9%. X-ray diffraction on the bulk specimens produced with composition 52 mol % Nb (near the Nb_2O_5-Al_2O_3 eutectic) has indicated that there exists a substantial quantity of crystalline phase, which is consistent with the phase diagram: $AlNbO_4$ is the equilibrium phase predicted at 50 mol. % Nb_2O_5. Further analysis on other compositions is in progress.

Results to date indicate that a liquid phase forms in Nb_2O_5-Al_2O_3 powder compacts at elevated temperatures (~1400ºC) and remains amorphous during cooling, consistent with the presence of a eutectic at approximately 1400ºC [1]. Indications that the liquid phase which forms at elevated temperature is not crystalline include the lack of detection with x-ray diffraction, and the unique contrast in the SEM (Figure 1). There exists evidence in the literature that Nb_2O_5 is a glass former in the presence of certain other oxides [2]. The secondary phase observed in Figure 1 wets alumina.

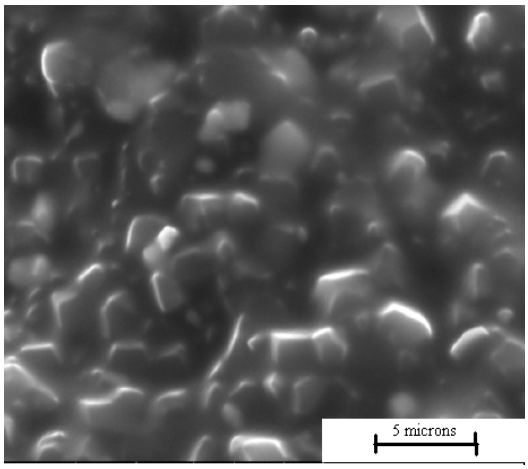

Figure 1. Scanning electron micrograph of a composite formed by mixing 80 vol. % Nb with 20 vol. % Al_2O_3. It is likely that the lack of contrast observed around the grains (both Al_2O_3 and Nb) in the microstructure is caused by the presence of a thin amorphous layer which is predicted to be a Nb_2O_5-based glass.

Future work will focus on microstructural and structural analyses to determine the relative amounts of glassy phase present. For select compositions, thermal analysis will be employed to measure the glass transition temperature if appropriate. Finally, bonds will be formed between porous and dense alumina for mechanical testing.

REFERENCES

1. R. S. Roth, J. R. Dennis, and M. F. Howard, Phase Diagrams for Ceramists, American Ceramic Society, Inc., Volume 6, pp155 (1987).

2. Doremus, RH (1994) **Glass** Science, 2nd Edition. John Wiley & Sons, New York, 339 pp. ISBN 0471891746